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Image Analysis Used to Count and Measure Etched Tracks from Ionizing Radiation

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ABSTRACT

We have developed techniques to use digitized scanning electron micrographs and computer image analysis programs to measure track densities in lunar soil grains and plastic dosimeters.

Tracks in lunar samples are formed by highly ionizing solar energetic particles and cosmic rays during near surface exposure on the Moon. The track densities are related to the exposure conditions (depth and time). Distributions of the number of grains as a function of their track densities can reveal the modality of soil maturation. We worked on two samples identified for a consortium study of lunar weathering effects, 61221 and 67701. They were prepared by the lunar curator's staff as polished grain mounts that were etched in boiling 1 N NaOH for 6 h to reveal tracks. We determined that backscattered electron images taken at 10% contrast and ~50% brightness produced suitable high contrast images for analysis. We used the NIH Image program to cut out areas that were unsuitable for measurement such as edges, cracks, etc. We ascertained a gray-scale threshold of 25 to separate tracks from background. We used the computer to count everything that was two pixels or greater in size and to measure the area to obtain track densities. We found an excellent correlation with manual measurements for track densities below 1x10⁸ cm⁻². For track densities between 1x10⁸ cm⁻² to 1x10⁹ cm⁻² we found that a regression formula using the percentage area covered by tracks gave good agreement with manual measurements. We determined the track density distributions for 61221 and 67701. Sample 61221 is an immature sample, but not pristine. Sample 67701 is a submature sample that is very close to being fully mature. Because only 10% of the grains have track densities less than 10° cm⁻², it is difficulty to determine whether the sample matured in situ or is a mixture of a mature and a submature soil.

Although our analysis of plastic dosimeters is at an early stage of development, results are encouraging. The dosimeter was etched in 6.25 N NaOH at 70°C for 16 h. We took 200x secondary electron images of the sample and used the NIH Image software to count and measure major and minor diameters of the etched tracks. We calculated the relative track etch rate from a formula that relates it to the major and minor diameters. We made a histogram of the number of tracks versus their relative etch rate. The relative track etching rate is proportional to the linear energy transfer of the particle. With appropriate calibration experiments the histogram could be used to calculate the radiation dose.

INTRODUCTION

Counting and measuring etched tracks from ionizing radiation can be used for various scientific purposes such as to determine radiation exposure ages and radiation dosage. Last summer we showed that computer image analysis could be use for counting tracks. This summer we continued to refine this process and we used image analysis to make morphological measurements of tracks in plastics that are used to determine radiation dose.

Price and Walker (1962) discovered that very ionizing radiation, such as fission fragments and cosmic rays, produces a trail of damage in dielectric materials that can be etched with a reagent to form visible tracks (cf. Fleischer et al., 1975; Durrani and Bull, 1987). Their discovery has led to practical applications such as Nuclepore filter paper and cosmic ray dosimeters used by astronauts. Scientific applications include fission track dating of geological samples and cosmic ray-solar energetic particle weathering effects on lunar samples. From the beginning quantitative scientific results have followed from counting tracks on micrographs and by micrographically measuring track morphological characteristics. The sophistication and ready availability of image processing software can reduce this tedious labor.

Exposure of regolith grains to radiation and meteoroid impacts on the Moon, asteroids, some planets and satellites, and interplanetary dust particles produce measurable forms of "weathering.". Research has shown that these measurable effects correlate in lunar soils (McKay et al., 1991). Nevertheless, the correlations are very crude because the weathering effects on the Moon are usually measured as a bulk average for a given soil. Most weathering measurements are not very useful for making quantitative predictions of exposure age or even giving a relative measure of maturity for the soil. Furthermore, regolith soils mature by at least two distinct processes: by in situ weathering and by mixing. Bulk average measurements cannot distinguish the maturation processes. To improve our understanding of space weathering, we should find these correlations on a grain by grain basis. During the ASEE summer program, we concentrated principally on one form of weathering, namely the formation of tracks in individual soil grains.

Humans in space are exposed to a much more severe radiation environment than workers in nuclear plants. In low earth orbit the exposure is primarily from the inner Van Allen belt of trapped solar radiation. This is especially harsh in the South Atlantic Anomaly where the belt dips lower in altitude than on average. Consequently it is very important to monitor exposure to radiation for humans in space. One method to do this is to have astronauts wear personnel dosimeters. One form of dosimeter that is used is a sandwich of plastic sheets which record tracks of ionizing radiation.

Etching lunar soil grains and plastic sheets in a suitable reagent reveals tracks by producing pits at the track locations. We used a scanning electron microscope (SEM) to make digital images of the etched surfaces of polished grain mounts and the plastic sheets. We developed procedures to rapidly measure track densities with image processing software.

The refinement of track counting techniques will be used to study the maturation of lunar soils as part of a consortium study of various maturation effects. We worked on lunar samples 67701 and 61221 that have been selected by the consortium for the study of lunar soil maturation effects.

We also began exploring the use of computer image analysis to count and to measure diameters of etched tracks in plastics. The major and minor diameters of the tracks are related to the linear energy transfer (LET) of the ionizing particle. By measuring the LET of each track we can add them to find the radiation dose.

Using computer analysis to measure tracks will reduce the tedious measurement of manually counting tracks or of individually measuring them microscopically. This will make track measurements a much more useful tool for understanding radiation environments.

MATURATION MODES OF LUNAR SOILS 61221 AND 67701

EXPERIMENTAL DETAILS

Samples 61221 and 67701 had been sieved into various size fractions by David McKay's group. We had several hundred grains from the 90-150 µm size fraction prepared by the lunar curator's staff as a polished grain mount. The grain mount was etched in boiling 1 N NaOH solution for six hours. The grain mount was rinsed, dried, and coated with ~10 nm layer of AuPd to conduct the electron beam to ground. Most of this work had been done before the beginning of the summer program.

We obtained images on an ISI SEM. The sample was oriented perpendicular to the electron beam. The same condenser lens setting and aperture were used for all images. Nevertheless, the microscope is not equipped with a Faraday cup and we could not be sure of reproducing the same beam current exactly for each microscope session. The working distance knob was set at 8 mm, the focus knobs were set at 5 turns clockwise, and the image was brought into focus initially by adjusting the sample height. This procedure assures that magnification and resolution will be consistent from one session to another.

We determined magnification calibration with a stage micrometer. Last year we verified that it remained consistent within 1.5%. The SEM is capable of making conventional secondary electron images (SEI) and it is also equipped with a back-scattered electron (BSE) detector. Secondary electrons produce a gray scale micrograph that looks very much like a regular black and white photograph. If SEI were used, we felt that fairly sophisticated image processing would be necessary to use the computer to distinguish tracks from background. BSE images, however, naturally showed a high contrast between tracks and background. We purposely chose to exploit this property and took digital images that appeared to the naked eye to be almost binary with very little gray. Using the computer we could set the contrast and brightness to numerically reproducible settings. From our experience from last year, we thought that we could use the same contrast and brightness settings. However, last year's sample had been etched for 15 h. Consequently its tracks were much larger. When we used the contrast and brightness settings from last year, we greatly undercounted the sample. This meant that we had to search again for the proper settings. We chose a wide variety of contrast settings and adjusted brightness settings visually to reproduce a high contrast image. We chose to work at a contrast setting of 10%. The brightness settings ranged from 48.75% to 50.55%. At any one session the range was much smaller, but the range from session to session was greater because the beam current was not exactly reproducible.

We produced digital images using an eXL computer manufactured by Oxford Instruments, formerly Link Analytical. The computer has a proprietary operating system and software. The system is designed to be used with electron microscopes and it controls energy dispersive x-ray analysis as well as digital imaging. Digital images were collected as a Kalman average for 90 s. The images were 512 x 512 pixels at a 256 gray-scale (8 bit). We primarily worked at a magnification of 10000x, but we worked as low as 5000x for grains with low track densities. The images were up loaded to the building 31 network (node: SN-Titan). From the building 31 network the files were downloaded to a UHCL Macintosh (node: regolith).

The images were analyzed using NIH Image software, a public domain program. This software is faster than the eXL software and we do not effectively tie up the SEM doing our analyses. We created a mask for each image to obscure parts of the image we did not wish to analyze such as areas off the edge of the grain, large cracks, etc. We set a binary threshold at a gray scale of 25, and had the computer count everything that had a size of two pixels or greater. NIH Image also returned the total area covered by tracks.

CALIBRATION

Figure 1 shows a correlation diagram of track density measurements using image analysis with conventional measurements from a photomicrograph. The correlation is excellent for track densities below 1x10⁸ cm⁻². However, above track densities of

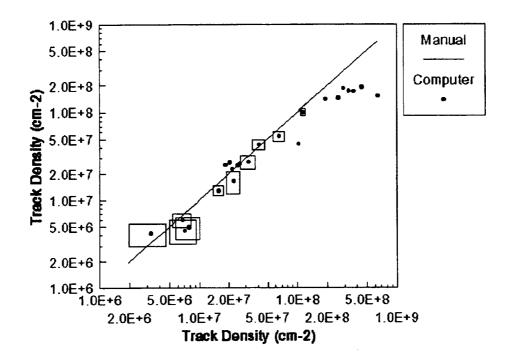


Figure 1. Graph of track densities in lunar soil grains from samples 61221 and 67701. The ordinate has values determined from counts using computer software. The abscissa has values determined by manual counting. Rectangles represent one standard deviation uncertainty in the measurements and are shown only for a representative set of points.

1x10⁸ cm⁻² the image analysis technique shows saturation as we had found last year. We found that the area covered by the tracks was proportional to the number of tracks. We calculated a regression line that correlates the percentage area of the tracks with the track density. In Fig. 2 we show a graph of manually counted track density versus track density determined using percentage area and the regression equation. When we present histograms of the two samples we indicate which measurements result from computer counting, which from the area regression line, and which were equivalent using both methods.

RESULTS

Track density distributions for samples 61221 and 67701 are shown in Figs. 3 and 4. Blanford et al. (1979) (or McKay et al., 1991) have discussed the relationship of the track density distribution to the modality of soil maturation. The two figures clearly show that the two soils have different radiation histories. Soil 61221 is immature which means that most of its soil grains have not been exposed for very long (<~10⁴ y) within the top millimeter of the lunar surface. That there are a relatively small number of grains with

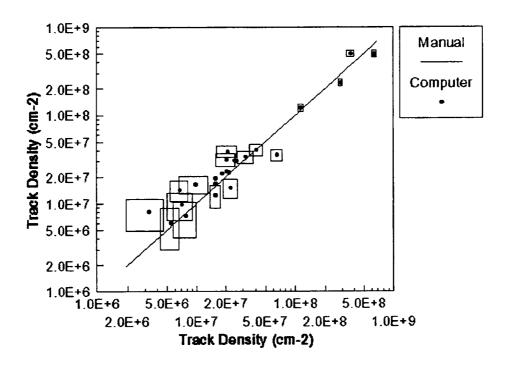


Figure 2. Track densities determined from the percentage area of the image covered by tracks versus track densities determined from manual counting. Rectangles represent one standard deviation uncertainty in the measurements and are shown only fro a representative number of points.

track densities $<10^7 \text{cm}^{-2}$ indicates that this soil has been within ~ 1 cm of the lunar surface for a relatively long time ($\sim 10^6$ y). Soil 67701 is submature, but nearly mature. Because only 10% of the soil grains have track densities $<10^8$ cm⁻², it is difficult to tell whether the soil is bimodal consisting of two mixed components (submature type II) or it has matured in situ (submature type I). The maturity designations for 61221 and 67701 agree with the designations from the ferromagnetic resonance index I₂/FeO. It is known that the darkening of the albedo of lunar soils with increased maturity is an effect that is dominated by the smallest fractions of the lunar soil (Fischer et al., 1994). We plan to repeat these measurements with the 10-20 μ m and 20-45 μ m fractions to determine whether or not these smaller fractions give qualitatively the same information as the 90-150 μ m fraction.

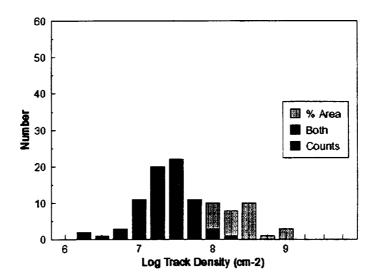


Figure 3. Solar flare and cosmic ray track density distribution for the 90-150 µm fraction of lunar soil 61221 (102 grains). Measurements determined by computer counting alone are shown as solid, measurements determined by percentage area regression are shown by the lightest shading, and measurements that were the same for both methods are shown by the intermediate shading.

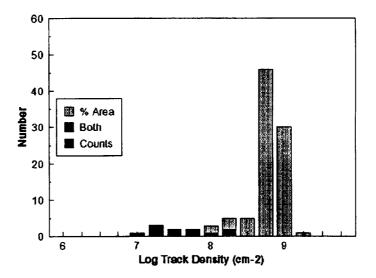


Figure 4. Solar flare and cosmic ray track density distribution for the 90-150 μ m fraction of lunar soil 67701(98 grains). Measurements determined by computer counting alone are shown as solid, measurements determined by percentage area regression are shown by the lightest shading, and measurements that were the same for both methods are shown by the intermediate shading.

ANALYZING PLASTIC TRACK DOSIMETERS USING IMAGE ANALYSIS

EXPERIMENTAL DETAILS

Experimental work for this project is at a very preliminary stage. A personnel dosimeter that had been flown on STS 59 was provided by Dr. Gautum Badwhar. It contained 4 sheets of CR-39 plastic (allyl diglycol carbonate) interleaved with thin sheets of Makrofol (bisphenol-A polycarbonate). We etched part of one of the CR-39 sheets in 6.25 N NaOH at 70°C for 16 h. We coated the sample with ~10 nm AuPd for electron microscopic observation.

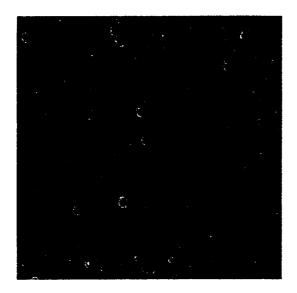
We took both SEI and BSE images with varying contrast and brightness. In this case the SEI images proved to be the most useful. Because we cannot set the contrast and brightness of SEI images with the digital accuracy of the BSE images, we took a series of micrographs using various settings that gave about the same visual appearance. We found that there needs to be some adjustment of the threshold for image analysis, but the counting and measuring were not strongly dependent on the conditions used. This should lead to very reproducible results. However, more experimentation will be necessary to establish conditions reliably.

Figure 5 shows a 200x SEI image of the etched CR-39 dosimeter with the counting record on the right. Besides counting the tracks the NIH Image software also can measure the major and minor diameters of the tracks. Durrani and Bull (1987) give the following formulas for the major diameter D and the minor diameter d

$$D = \frac{2h(V^2 - 1)^{\frac{1}{2}}}{V \sin \theta + 1}$$

$$d = 2h \left(\frac{V \sin \theta - 1}{V \sin \theta + 1} \right)^{1/2}$$

where h is the amount of surface removed during etching, V is the relative etch rate V_T/V_B , and θ is the angle the track makes with the surface of the plastic. V_T is the etch rate along the track and V_B is the bulk etch rate. The relative etch rate V is the critical measurement for dosimetry because it is directly related to the linear energy transfer (LET) of the particle (Price et al., 1967; O'Sullivan et al., 1971). Manipulating the formulas for d and D, one finds that the relative etch rate V is given by



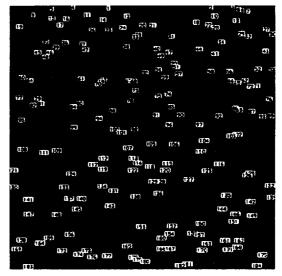


Figure 5. On the left is a secondary electron image of an etched CR-39 plastic dosimeter exposed on mission STS 59. The image on the right shows the tracks that were counted.

$$V = \sqrt{1 + \frac{D^2}{h^2 \left[1 - \left(\frac{d}{2h}\right)^2\right]}}$$

Normally h is measured during each experiment, but we did not plan far enough ahead and did not make this measurement. We estimated it instead from He and Price (1992) as being 21 μ m for our etching conditions. Figure 6 shows a histogram of the number of tracks as a function of their relative etching rate. Because the relative etching rate is directly related to LET, if we had a calibration for this plastic, the dose could be calculated by multiplying the number in each column by the appropriate LET for that column and adding them together. Most of the tracks represented in this histogram are probably protons and α -particles. However, the three tracks on the right of the histogram are very likely heavier ions. Many images would have to be analyzed to include these heavy ions with good statistical accuracy and thereby give a good measure of the dose.

CONCLUSION

During this summer program we have found that using image analysis we can count and measure tracks with as great a reliability as by using manual means. We also found that changing etching conditions changes image analytical conditions. Establishing the correct analytical conditions is quite time consuming. For time efficient measurements one must first establish the desired etching conditions and then use them consistently for

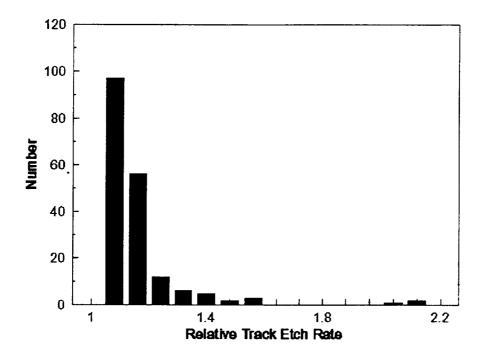


Figure 6. A histogram of the number of tracks as a function of their relative etching rate. The relative etch rate is directly related to the linear energy transfer (LET). By multiplying the number of tracks by the appropriate LET the dose can be obtained.

future work. For working with plagioclase grains in lunar samples we now have established two sets of image analytical conditions for two etching conditions. We are now able to analyze lunar plagioclase samples rapidly for the two cases.

We have established preliminary conditions for counting and measuring major and minor track diameters in CR-39 plastic. We have not yet optimized these conditions. Furthermore the measurement of major and minor track diameters is not the most accurate method of measuring the relative track etch rate V. It is better to use the track length to obtain V (Durrani and Bull, 1987). This requires measuring the track's projected length and its depth. Scanning electron micrographs are not suitable for this, whereas transmission optical micrographs are. Work will continue to try to do these measurements with digital images taken on a stereo optical microscope. If the goal of these measurements were to measure the charge-energy spectrum of cosmic rays, then measuring track lengths would be necessary. However, for space dosimetry the measurement of diameters may be adequate because most of the ionizing particles are Proton tracks are small and diameter measurements may be adequate. protons. Nevertheless, the heavy ions make a significant contribution to the total dose even though they are small in number. This is the result of the LET being proportional to the square of the atomic number. At this time we can not definitively answer whether we can simply measure the major and minor diameters or if we must also measure lengths and depths.

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Unified Approach For Incompressible Flows

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